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The Effect of Processing Parameters on the Microwave Absorption by Polyaniline/PMMA Composites

Darren A. Makeiff

Defence R&D Canada – Atlantic

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DRDC Atlantic TM 2004-301
January 2005

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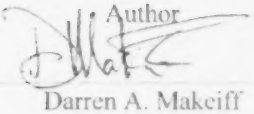

Darren A. Makeiff

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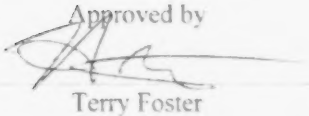

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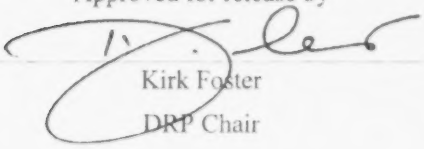

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Abstract

Conductive thermoplastic PMMA composites containing Polyaniline conductive filler were prepared. Conditions were varied in order to examine their effect on the electrical and microwave properties. Ball-milling for 2 h prior to heating at 175 °C at 80 psi for 3 min in the presence of 10 wt % hydroquinone plasticizer was found to give composites with the highest measured microwave absorption in the X-band (8-12 GHz). Loss tangents as high as 44 were attained under these conditions.

Résumé

On a préparé des composites conducteurs à base de poly(méthacrylate de méthyle) (PMMA), une matière thermoplastique, et renfermant une matière de charge conductrice, de la polyaniline. On a fait varier les conditions expérimentales afin d'étudier leurs effets sur les propriétés électriques et hyperfréquences. On a montré qu'un broyage avec des boulets pendant 2 heures, suivi d'un chauffage à 175 °C et sous une pression de 80 lb/po^2 en présence de 10 % en poids d'hydroquinone (plastifiant), permettait d'obtenir les composites ayant l'absorption micro-onde la plus importante dans la bande X (8-12 GHz). Des facteurs de perte électrique allant jusqu'à 44 ont été obtenus dans ces conditions.

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Executive summary

Introduction

Effective Radar Absorbing Materials (RAM) must possess conductive networks in order to attentuate incident electromagnetic (EM) radiation and minimize reflection. This is typically achieved by incorporating a finely dispersed conductive filler material within an insulating matrix polymer possessing good mechanical properties. Conducting polymers are favorable conductive filler materials because of their cheap synthesis, controllable conductivity, lightweight, and environmental stability. We have previously demonstrated that composites containing doped polyaniline (PAni) can be incorporated into polymer composites and show good potential as RAM.

Results

The effect of various processing parameters in the formation of conductive polymer composites containing doped PAni conductive filler were investigated in detail. Varying processing conditions such as mixing time, temperature, heating time, and wt % plasticizer (HQ) had significant effects on the electrical (i.e., DC conductivity) and microwave properties (i.e., real and complex permittivities and loss tangent). Processing conditions were optimized to give composites with maximum microwave absorption.

Significance

The significance of the findings in this study are that the materials described here can be synthesized cheaply, and the desired properties can be controlled. The control of properties such conductivity, complex permittivity, and the loss tangent are necessary in order to design effective RAM.

Future plans

Future investigations may include:1) scaling up the process described, 2) incorporation of other conducting polymers or other doped PAnis as conductive fillers into the same matrix, 3) incorporating the composites reported here into layered materials, 4) measurement of other microwave properties such as shielding effectiveness and reflectivity and to examine these properties at microwave frequencies other than the X-band (8-12 GHz).

Makeiff, D. A. 2004. The Effect of Processing Parameters on the Microwave Absorption by Polyaniline/PMMA Composites. DRDC Atlantic TM 2004-301, Defence R&D Canada – Atlantic.

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Introduction

Les matériaux absorbant les ondes radars (RAM) efficacement doivent contenir des réseaux conducteurs afin d'absorber le rayonnement électromagnétique (EM) incident et réduire au minimum sa réflexion. On obtient habituellement ces effets grâce à l'incorporation d'un matériau de remplissage conducteur finement dispersé dans une matrice polymère isolante ayant de bonnes propriétés mécaniques. Les polymères conducteurs sont de bons candidats comme matière de charge conductrice, en raison de leur coût de synthèse peu élevé, de leur conductivité contrôlable, de leur faible densité et de leur stabilité dans l'environnement. Nous avions déjà montré que des composites renfermant de la polyaniline (PAni) pouvaient être incorporés dans des composites polymères et présenter un bon potentiel comme RAM.

Résultats

On a étudié en détail les effets de divers paramètres de traitement lors de la formation de composites polymères conducteurs contenant de la PAni comme matière de charge conductrice. Divers paramètres de traitement, comme le temps de mélange, la température, la durée de chauffage et le % en poids de plastifiant (HQ) ont des effets importants sur les propriétés électriques (par exemple la conductivité du c.c.) et hyperfréquences (par exemple la permittivité réelle, la permittivité complexe et le facteur de perte électrique). Les conditions de traitement ont été optimisées afin d'obtenir des composites ayant une absorption micro-onde maximale.

Importance

Les résultats obtenus lors de cette étude sont importants, car ils montrent que les matériaux recherchés peuvent être synthétisés à faible coût et que les propriétés recherchées peuvent être contrôlées. Le contrôle de propriétés, comme la conductivité, la permittivité complexe et le facteur de perte électrique, est nécessaire afin de concevoir des RAM efficaces.

Projets

Les prochaines études pourraient comprendre : 1) passage à une échelle plus grande du procédé décrit; 2) incorporation dans la même matrice d'autres polymères conducteurs, autres que de la PAni ou des PAni dopées, comme matière de charge conductrice; 3) incorporation des composites obtenus dans des matériaux multicouches; 4) mesure d'autres propriétés hyperfréquences, comme l'efficacité de blindage et la reflectivité, afin d'étudier ces propriétés à des fréquences micro-ondes autres que celles de la bande X (8-12 GHz).

Makeiff, D. A. 2004. The Effect of Processing Parameters on the Microwave Absorption by Polyaniline/PMMA Composites. DRDC Atlantic TM 2004-301, Defence R&D Canada – Atlantic.

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This research was supported in part by the NSERC Visiting Fellowship in a Canadian Government Laboratory program. We would also like to thank Brent Gowen (Advanced Imaging Department, Biology, University of Victoria) for obtaining the light microscope sections of the composite samples used for this study.

1. Introduction

This report discusses the potential of polymer composites containing the conducting polymer (CP), Polyaniline (PAni) as Radar Absorbing Materials (RAM) in the X-band frequency range (microwave). One of the requirements in designing effective RAM are the presence of conductive networks.[1,2] Conductive networks are necessary to facilitate the dissipation of incident electromagnetic (EM) radiation, via displacement currents induced within the material by the electric field component of the incident wave.[1,2] The energy absorbed is thus dissipated as heat.[1,2]

CPs offer many advantages over traditional conductive fillers in microwave absorption due to their lightweight, tunable conductivity, cheap synthesis and processibility in a wide variety of matrices at low weight percent.[3] In contrast, traditional conductive fillers such as metals (i.e., iron and nickel) are heavy, while carbon-based fillers require high loading to achieve the desired conductivity. Microwave properties of PAni have been documented in the open literature.[4] The potential of conductive PAni/poly(methylmethacrylate) thermoplastics as RAM has recently been assessed based on our own measurements.[5,6]

In order to quantitatively assess a potential RAM, certain microwave properties such as the complex permittivity (ε) must be determined.[3] The complex permittivity (equation 1) consists of real and imaginary components (ε' and ε'', respectively), which arise from polarization and conductivity in a material, respectively.[7]

$$\varepsilon = \varepsilon' - i\varepsilon'' \qquad (1)$$

Knowledge of ε' and ε'' allows for the calculation and quantification of the amount of incident EM absorbed, reflected, and transmitted through a material. Note that ε'' is related to the microwave conductivity (σ_{mw}) by equation 2,

$$\sigma_{mw} = 2\pi f \varepsilon_0 \varepsilon'' = \sigma_{dc} + \sigma_{ac} \qquad (2)$$

where f is the frequency (in GHz), ε_0 is the permittivity of free space, and σ_{dc} and σ_{ac} are the dc and ac conductivities, respectively.

A good measure of microwave absorption the loss tangent ($tan\delta$), which is derived from ε'' and ε' (equation 3).[3] Materials with $tan\delta > 1$ are considered "lossy" materials,[3] while $tan\delta$ values in the order of 10 are considered to indicate strong absorption of radiation at a given frequency.[3]

$$tan\delta = \varepsilon''/\varepsilon' \qquad (3)$$

Preliminary data on the effect of various processing variables on the electrical properties of compression moulded PAni/PMMA composites has been reported.[6] The composites in this study were prepared using a procedure based on work reported by

Morgan and coworkers.[8] Huber and coworkers determined loss tangents between 1 and 3 for their PAni/PMMA composites.[6]

In this report, the processing conditions for PAni-pTsA/PMMA are investigated in greater detail than in the preliminary reports.[5,6] Sample mixing, melting temperature, heating time, amount of plasticizer, and amount of conducting polymer were varied and their effects on the electrical and microwave properties of PAni/PMMA composites are discussed here.

2. Experimental

2.1 General Experimental

Aniline (99.5%) was purchased from Aldrich chemicals and was distilled *in vacuo* prior to usage. *para*-toluene sulfonic acid (pTsA, 98 %), ammonium peroxydisulfate (APS) were used as purchased from Aldrich. Hydroquinone (HQ) was used as purchased from BDH Chemicals. Poly(methyl methacrylate) (PMMA, Buehler transoptic powder) was used as purchased from Buehler Canada.

2.2 Polyaniline Synthesis

PAni-pTsA emeraldine salt (Figure 1) was synthesized via the conventional oxidative polymerization of aniline with APS. Aniline was added to ethanol or H_2O solutions of 1 M pTsA. Reactions were conducted at different temperatures and were allowed to proceed for various lengths of time. The mixtures were equilibrated at temperatures ranging 0-25 °C prior to APS addition. After allowing the reactions to proceed for an appropriate amount of time, they were filtered, washed thoroughly with ethanol and acetone, and then dried for 24 h in vacuo to give PAni-pTsA as dark green powders ranging in conductivity from 0.5 to 6 S/cm.

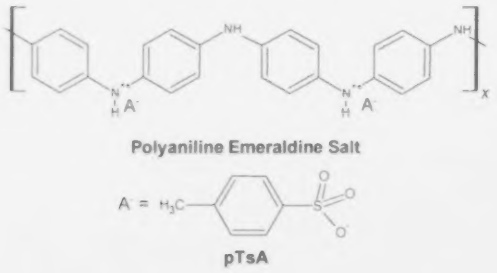

Figure 1. *Schematic representation of the structure of PAni emeraldine salt.*

2.3 Polyaniline-Poly(methyl methacrylate) Composite Synthesis

Conductive PMMA composites were synthesized as follows. PAni-pTsA, PMMA, varied amounts of HQ, and one acrylic ball were added to a polystyrene vial and shaken using a SPEX mixer/mill (catalogue # 8000) for varied lengths of time. The powder mixture was then transferred to the loading chamber of a Buehler Pneumet I mounting press, and placed under 80 psi of compression. The sample chamber was heated to appropriate temperatures for various times, followed by cooling the sample to room temperature using a cold water condensor. Samples were removed from the mounting press chamber as rigid plastic disks, which were carefully machined into a 10.16 × 22.86 mm rectangle for DC conductivity (σ_{dc}) and permittivity measurements.

The Pneumet I mounting press used in this study was not designed for variable temperature experiments. The heating assembly consists of a separate heating mantle, which surrounds the sample chamber, which is preset by the manufacturer to heat samples to only 160 °C. However the heating output can be adjusted manually by turning a screw inside the mantle electrical box. Two heating mantles were used in this study; one set at the manufacturers setting was used to calibrate heating temperature and the other was used for variable temperature runs. Sample temperatures were recorded using a thermocouple, which was inserted into the top of the mounting press. However, the temperature measured in this position (~110 °C) was found to be lower than the manufacturers setting (160 °C). Therefore sample temperature measured for runs at temperatures above the manufacturers setting were corrected accordingly. This was not realized in earlier reports.[5,6,9]

2.4 Instrumentation and Measurements

The DC conductivity (σ_{dc}) for PMMA composites could not be determined using the four point probe method because of high contact resistances. Values for σ_{dc} were determined by applying silver paint to the short ends of the rectangular samples and measuring the resistance (R) down the length of the sample. The DC conductivity can be calculated using equation (4):

$$\sigma_{dc} = L/R \times A \qquad (4)$$

where, A is the area of each silver paint electrode (in cm^2).[10] Values for σ_{dc} were similar to values for the microwave conductivities (σ_{mw}) measured at 10 GHz.

Permittivity measurements from 8 to 12 GHz were made using an HP8720C vector network analyzer via the transmission-reflection method.[11]

3. Results and Discussion

3.1 The Effect of Mixing Time

Interparticle contacts play an important role in influencing the overall conductivity of a composite.[7] Smaller particles generally result in greater conductivity over larger particles because of the greater surface-to-volume ratio.[7] Ball-milling is one method of dispersing powder samples as well as reducing powder particle size. Therefore we examined the effect of ball-milling on σ_{dc}, ε', ε'', and $tan\delta$ for our composites containing PAni-pTsA. Powder mixtures were ball-milled for various lengths of time prior to compression molding (80 psi, 125 °C, 9 wt % PAni-pTsA, 7.5 wt % HQ).

Figure 2 shows the effect of mixing via ball-milling on the microwave dielectric properties of PAni-pTsA/PMMA composites. The sharpest increase in microwave absorption occurs for samples that were ball-milled between 1 and 120 min. Both ε' and ε'' increase linearly during this time. The increase is more significant for ε'' (i.e., 5-fold), which contributes most to the increase in $tan\delta$.

A light microscope image of a microtomed section of PAni-pTsA/PMMA is shown in Figure 3. Web-like networks of PAni-pTsA (black regions) can be clearly seen between the melted and compressed PMMA matrix particles. The web-like structures are microns in width and interconnect larger PAni-pTsA masses dispersed in the PMMA matrix. During the first 120 min of ball-milling, PAni-pTsA particle size is probably greatly reduced (i.e., to microns) relative to PMMA (i.e., tens of microns). Smaller PAni-pTsA particles probably pack more efficiently in the void spaces between PMMA particles when compressed, thus reducing the number of breaks in the web-like structures (Figure 3) and larger, poorly dispersed PAni-pTsA masses. Ball-milling for greater than 120 min probably does not change the PAni-pTsA particle size significantly. PMMA particles were not altered after ball-milling.

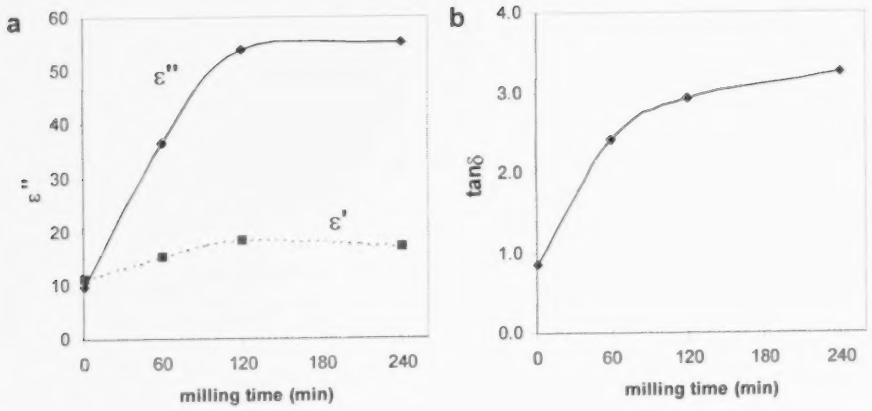

Figure 2. *The effect of ball-milling time on ε'', ε', and tanδ (10 GHz). Samples contained 10 wt % hydroquinone and were heated at 160 °C for 3 min.*

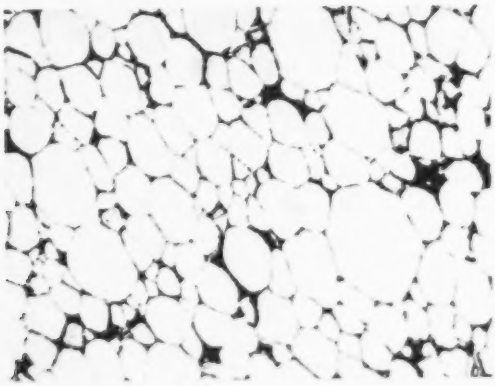

Figure 3. *Light microscope image (200×) of a microtomed section from a PAni-pTsA/PMMA composite.*

3.2 The Effect of Temperature

The glass transition temperature (T_g) and melting temperature (T_{melt}) for the PMMA used in this study (Buehler transoptic powder) were determined to be ~110 and 154 °C, respectively, by differential scanning calorimetry. Therefore composite powder samples were compression molded above 160 °C.

Table 1 shows values for ε', ε'', σ_{dc}, and $tan\delta$ determined for PAni-pTsA/PMMA composites produced at various temperatures. Two sets of data are shown for composites prepared under slightly different conditions. For both runs, the maximum $tan\delta$ occurs near 175 °C, with ε'' at a maximum and ε' at a minimum. Below 175 °C, the lower conductivity may be attributed to incompletely fused PAni-pTsA particles. The lower conductivity and ε'' for composites processed above 175 °C may be due to decomposition of PAni-pTsA. PAni-pTsA is known to decompose above 200 °C.[8]

Table 1. *ε', ε'', σ_{dc}, and tanδ (10 GHz) for PAni-pTsA/PMMA composites prepared at different temperatures.*

	T (°C)	σ_{dc} (S/cm)	ε''	ε'	$tan\delta$
PAni-pTsA1[a]	160	-	24.4	9.96	2.4
PAni-pTsA1[a]	175	0.438	96.3	6.00	16.0
PAni-pTsA1[a]	197	0.149	22.4	13.30	1.7
PAni-pTsA1[a]	220	0.237	49.5	9.70	5.1
PAni-pTsA2[b]	160	-	157.0	5.50	28.5
PAni-pTsA2[b]	175	-	182.0	4.13	44.1
PAni-pTsA2[b]	195	-	128.0	9.80	13.1

PAni-pTsA1 and 2 refer to two different batches of synthetic PAni-pTsA. [a]Samples contained 7.5 wt % hydroquinone, were ball- milled for 60 min, and were heated for 3 min. [b]Samples contained 10 wt % hydroquinone, were ball-milled 120 min, and were heated for 3 min.

3.3 Effect of Heating Time

The effect of heating time on ε'', ε', and $tan\delta$ at 160 and 175 °C, is shown in Figure 4. The loss tangent ($tan\delta$) increases with heating time up to ~3 min, where a maximum value is reached. After 3 min., $tan\delta$ values decline. Heating time only significantly effects ε'' (i.e., conductivity); $tan\delta$ mirrors ε'', while ε' is relatively constant. Before 3 min, PAni-pTsA particles may not completely fused, while beyond 3 min they begin to decompose. Near 3 min the PMMA particles probably fuse with each other reducing the surface area and density of conductive paths. The results are similar at both 160 and 175 °C, except the latter gave slightly higher $tan\delta$ due to slightly lower ε' (*vide supra*).

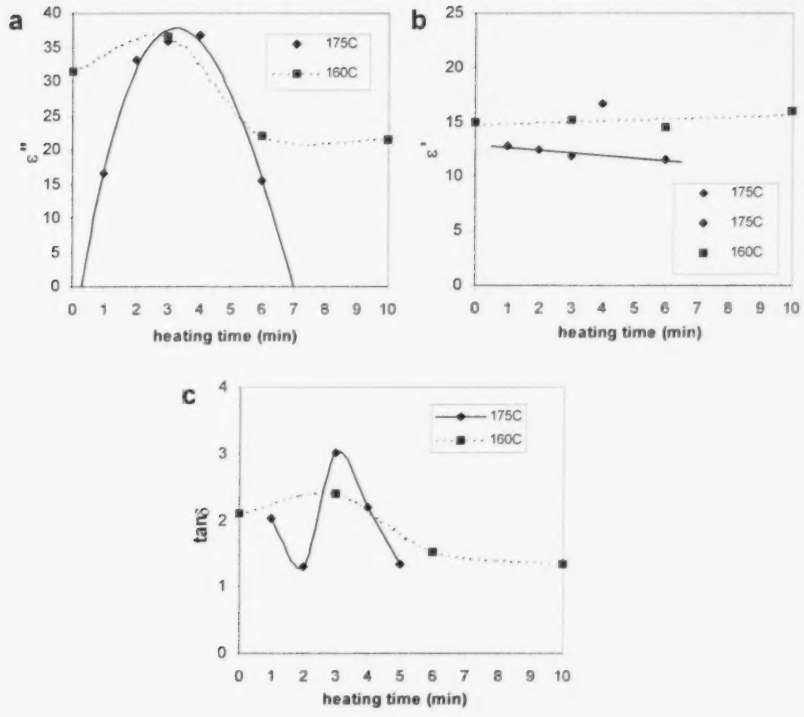

Figure 4. *The effect of heating time on a) ε'', b) ε', and c) tanδ at 160 and 175 °C. Samples that were heated at 160 °C contained 10 wt % hydroquinone and were ball-milled 120 min. Samples that were heated at 175 °C contained 7.5 wt % HQ and were ball-milled 30 min.*

3.4 The Effect of Plasticizer

Dihydroxybenzenes, such as hydroquinone (HQ) are known to help increase the conductivity of PAni-pTsA via "secondary doping".[12] Hydrogen bonding and π-stacking interactions can occur between secondary dopant and PAni-pTsA chains as well as with the counter-ions (i.e., primary dopant, or pTsA anions in this study). Morgan and coworkers reported that melt blended PAni-pTsA and PAni-DBSA/PMMA thermoplastics containing 7.5 wt % HQ showed the highest conductivity at the percolation threshold of each (15 and 20 wt %, respectively).[8] Therefore, we decided to check this for our composites as well.

Increasing the wt % HQ in PAni-pTsA/PMMA composites improves *tanδ* significantly (Figure 5). The complex permittivity (ε'') increases nearly an order of magnitude upon

increasing HQ from 0 to 10 wt % in the presence of 9 wt % PAni-pTsA. This is contrary to the work of Morgan and coworkers who reported that conductivity declined above 7.5 wt % HQ.[8] However, their samples contained 15 wt % PAni-pTsA.[8] Here, ε' slightly decreased with increasing wt % HQ. 10 wt % HQ was determined to be optimum (i.e., 1 : 1 PAni-pTsA : HQ by weight); no further increase for $tan\delta$ was observed above 10 wt % HQ. The wt % HQ for maximum $tan\delta$ probably depends on the wt % PAni-pTsA present in the composite (i.e., the ratio HQ : PAni-pTsA), however this was not determined experimentally.

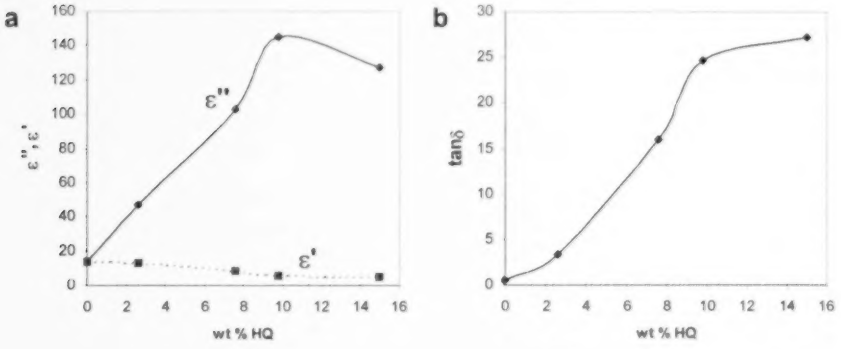

Figure 5. *Effect of wt % hydroquinone on a) ε'', b) ε', and c) $tan\delta$ at 10 GHz. Samples were ball-milled for min and heated at 200 °C for 3 min.*

3.5 The Effect of Weight % Polyaniline

The data presented in sections 3.1-3.4 suggests that the optimum conditions for compression moulding PAni-pTsA/PMMA thermoplastic composites are the presence of 10 wt % HQ, 2 h ball-milling, and heating at 175 °C for 3 min. Using these optimized conditions, we examined the microwave properties with varying wt % PAni-pTsA. The results are displayed in Figure 6. Note that the wt % PAni-pTsA investigated in the composites here was well above the percolation threshold.[9] The loss tangent ($tan\delta$) increases over the whole wt % range of PAni-pTsA investigated. Below 7 wt %, $tan\delta$ increases gradually, while above 7 wt % $tan\delta$ increases sharply. The sharp increase above 7 wt % is primarily due to a sharp drop in ε'. The complex permittivity (ε'') increases linearly between 0 and 10 wt %.

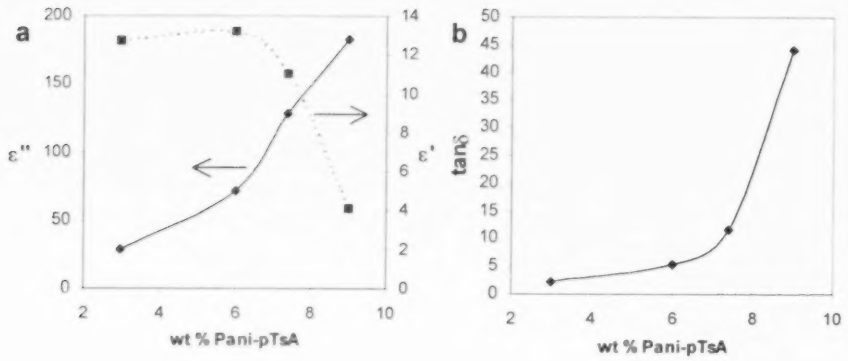

Figure 6. *Dielectric properties of PAni-pTsA/PMMA composites processed under optimized conditions. a) ε' and ε''. b) tanδ. Samples contained 10 wt % hydroquinone, were ball-milled for 120 min, and heated at 175 °C for 3 min.*

Five different PAni-pTsA/PMMA composites were prepared from PAni-pTsA samples synthesized in different batches and their electrical and the corresponding microwave properties were determined. The PAni-pTsA samples used had different σ_{dc}s, ranging from 0 to 6 S/cm,. Figure 7 shows that the dielectric properties of composites processed under the optimized conditions can be correlated to the PAni-pTsA pellet σ_{dc} used. A good linear relationship ($r^2 = 0.989$) is apparent between $tan\delta$ and pellet σ_{dc}. This is useful for preparing PAni-pTsA/PMMA composites with specific requirements from a given batch of synthetic PAni-pTsA. Similar trends can probably be established for PAni doped with other acids using the same set of processing conditions.

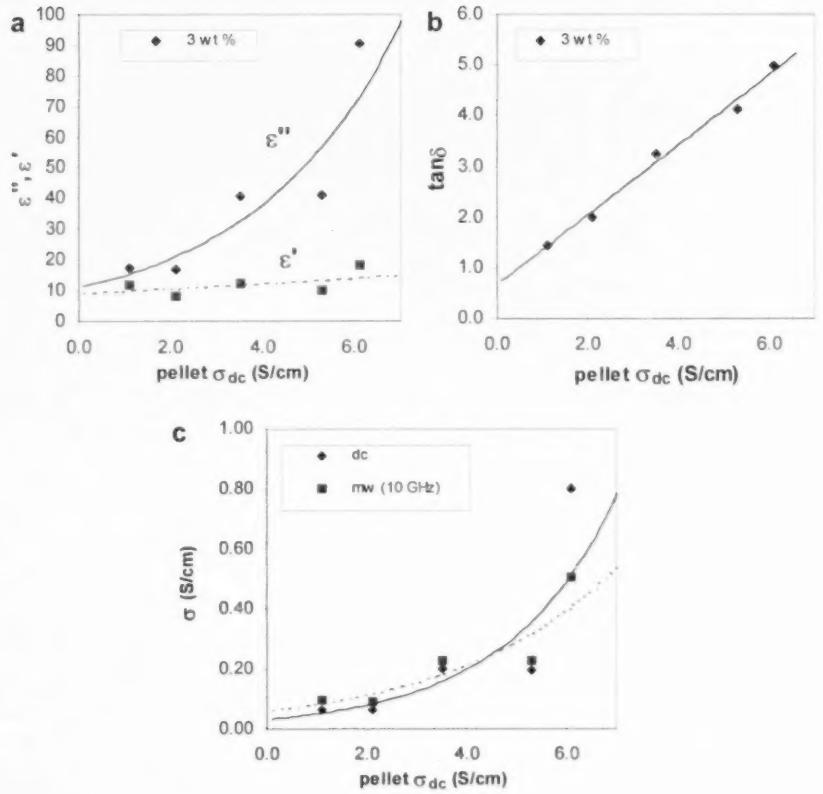

Figure 7. *The effect of PAni-pTsA pellet conductivity on a) ε' and ε'', b) tanδ, and c) σ_{dc} and σ_{mw} for PAni-pTsA/PMMA composites (3 wt % PAni-pTsA). Samples contained 10 wt % hydroquinone, were ball-milled for 120 min, and heated at 175 °C for 3 min.*

4. Conclusions

To summarize, we have demonstrated that the optimized conditions for synthesizing PAni-pTsA/PMMA composites for microwave absorption are 10 wt % HQ, 120 min ball-milling, and heating at 175 °C for 3 min at 80 psi. These conditions yield exceptionally lossy materials, with $tan\delta$ values ranging as high as 45; a 15-fold improvement over earlier attempts.[5,6] A good correlation was also found between σ_{dc} and the measured microwave properties, which is useful for producing PAni-pTsA/PMMA composites with desired properties from a given batch of synthetic PAni-pTsA. Future work may involve acquiring similar data for composites containing PAni doped with other acids, melt processing doped PAni/PMMA using other equipment, fabricating thinner composites (thickness = 1 mm here), and incorporating doped PAni/PMMA into multi-layered composites for broad-band microwave absorption.

5. References

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List of symbols/abbreviations/acronyms/initialisms

APS	ammonium peroxydisulfate
DBSA	*para*-dodecylbenzenesulfonic acid
ε	complex permittivity
ε_0	permittivity of free space
ε'	real permittivity
ε''	imaginary permittivity
EM	electromagnetic
f	frequency (in gigahertz, GHz)
HQ	hydroquinone
σ_{ac}	ac conductivity
σ_{dc}	dc conductivity
σ_{mw}	microwave conductivity
PAni	Polyaniline
PMMA	poly(methyl methacrylate)
pTsA	*para*-toluene sulfonic acid
RAM	radar absorbing material
tanδ	loss tangent

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14. ABSTRACT

(U) Conductive thermoplastic PMMA composites containing Polyaniline conductive filler were prepared. Conditions were varied in order to examine their effect on the electrical and microwave properties. Ball–milling for 2 h prior to heating at 175 °C at 80 psi for 3 min. in the presence of 10 wt % hydroquinone plasticizer was found to give composites with the highest measured microwave absorption in the X–band (8–12 GHz). Loss tangents as high as 44 were attained under these conditions.

(U) On a préparé des composites conducteurs à base de poly(méthacrylate de méthyle) (PMMA), une matière thermoplastique, et renfermant une matière de charge conductrice, de la polyaniline. On a fait varier les conditions expérimentales afin d'étudier leurs effets sur les propriétés électriques et hyperfréquences. On a montré qu'un broyage avec des boulets pendant 2 heures, suivi d'un chauffage à 175 °C et sous une pression de 80 lb/po2 en présence de 10 % en poids d'hydroquinone (plastifiant), permettait d'obtenir les composites ayant l'absorption micro–onde la plus importante dans la bande X (8–12 GHz). Des facteurs de perte électrique allant jusqu'à 44 ont été obtenus dans ces conditions.

15. KEYWORDS, DESCRIPTORS or IDENTIFIERS

(U) RAM; conducting polymers; polyanaline